

NBS REPORT 9286

TWENTY-SIXTH PROGRESS REPORT

to

National Aeronautics and Space Administration

on

Cryogenic Research and Development

for

Period Ending June 30, 1967



J. S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS
BOULDER LABORATORIES
Boulder, Colorado



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TABLE OF CONTENTS

l.	Phys	ical Properties of Cryogenic Fluids
	1.0	General Comments
	1.1	Dielectric Constant of Solid Parahydrogen
	1.2	Refractive Index of Hydrogen
	1.3	TAB Code and Tables of Evaluated Data
	1.4	Equation of State for Parahydrogen
	1.5	Absorption of Thermal Radiation in Liquid Hydrogen 4
2.	Cryo	ogenic Properties of Solids
	2. ĺ	Thermal Conductivity of Solids
		2.1.1 General Comments
		2.1.2 Program Status
		Fig. 2.1.1
		Fig. 2.1.2
		Fig. 2.1.3
		2.1.3 Materials Properties Data Book
		Fig. 2.1.4
		Fig. 2.1.5
		Fig. 2.1.6
	2.2	Thermocouple Thermometry
		2.2.1 General Comments
		2.2.2 Program Status
		Fig. 2.2.1
		2.2.3 Summary
	2.3	Thermal Expansion
		2.3.1 General Comments
		2.3.2 Program Status
		Fig. 2.3.1
		Fig. 2.3.2
		Fig. 2.3.3
		2.3.3 Materials Properties Data Book
		Table 2.3.1 Linear Thermal Contraction
		and Coefficients of Linear
		Thermal Expansion
3.	Cons	sultation and Advisory Services
J.	3.0	General Comments
	3.1	Centaur Program
	5.1	3.1.1 Stratification and Pressurization30
		7
	2 2	3.1.3 Helium Facility Study
	3.2	NERVA Program
	3.3	Hydrogen Contamination

4.	Cryogenic Flow Processes		34
	4.0 General Comments		34
	4.1 Experimental and Analytical Transfer Line Cooldown		34
5.	Cryogenic Propellant Venting Under Low Pressure Condition	ons	35
	5.0 General Comments		35
	5.1 Accomplishments during Current Reporting Period as	nd	
	Status of Project		35

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1. Physical Properties of Cryogenic Fluids

1.0 General Comments

Personnel contributing during this period were D. E. Diller, R. D. Goodwin, W. J. Hall, M. C. Jones, R. D. McCarty, H. M. Roder, and B. A. Younglove.

1.1 Dielectric Constant of Solid Parahydrogen

Since the last report a few more data points were taken of the solid dielectric constant on the melting line up to a maximum density of 0.09650 gm/cm³. The equation for polarizibility now recommended is

with ρ in gm/cm³. This is only slightly different from that previously recommended. This gives an r.m.s. deviation of 0.026% from the measurements. Also the polarization can be represented as a linear function in temperature,

$$P = 1.006082 + 0.000019 \text{ T cm}^3/\text{gm}$$

with T in °K. This gives an r.m.s. deviation of 0.029%.

The dielectric constant likewise can be given as a linear function of temperature over the range of temperatures measured. It is

$$\epsilon = 1.222 887 + 0.004 500 T$$

where T is in 'K; this represents the data to within 0.085% r.m.s.

1.2 Refractive Index of Hydrogen

Experimental determinations of the temperature, density and composition dependence of the Lorentz-Lorenz function for hydrogen are continuing. Measurements on gaseous parahydrogen along isotherms at 35°, 50°, 60°, and 100°K have recently been completed. The limiting value of the Lorentz-Lorenz function at low densities is nearly independent of temperature in this range. Along each isotherm the function increases to a maximum with increasing density. The heights of the maxima range from 0.05% to 0.35% above the low density values and decrease rapidly with increasing temperature.

Measurements on saturated liquid parahydrogen from the triple point to the critical point are in progress. In order to measure absolute values of the liquid refractive index while retaining the high precision and accuracy of an interferometer, fringes must be counted while slowly heating the fluid to a temperature above critical and then decreasing the pressure to vacuum. This scheme was suggested several years ago [1] and has been found to be quite practicable. The value of (n-1) can easily be measured with an uncertainty of less than 2 parts in 10,000 using this method.

D.E.D.

1.3 TAB Code and Tables of Evaluated Data

To establish the deviation pattern for the TAB code programs, a series of "best" values is needed. We established a "best" PVT surface by fitting a Benedict-Webb-Rubin type equation to our isotherms from 50 to 100°K (para), all of the data surveyed by Woolley (normal), and the PVT measurements of Michels, et al. (normal). In this approach, statistical limits were obtained for error in the derived

R. J. Corruccini, NBS Technical Note 323 (1965).

properties. Woolley's derived properties fall within one standard deviation of the present values.

The comparison was conducted in three regions as follows:

Region 1 Low temperatures to 180°R (Monograph 94)

Region 2 180 to about 2700°R (essentially no dissociation)

Region 3 2700 to 5000°R (region of dissociation).

A set of representative isobars was selected: 1, 15, 50, 150, 500, 1500, and 4500 psia. Along each of these isobars for appropriate intervals of temperature, the values interpolated from the TAB code were compared with the "best" values; differences were then plotted with a mechanical plotter.

Checks have been completed in all three regions, and the following major errors have been found:

- 1. At temperatures just above 180°R and pressures above 1500 psia. Here the enthalpy differences increase to as much as 100 BTU/lb. with corresponding errors in the other variables. The reason is that the values given in TN-130 extend only to 1500 psia. The values of TAB code at higher pressures were extrapolated linearly from entries below 1500 psia.
- 2. Deviations between the PVT surface of AiResearch and that of Woolley. The deviations are as large as 1.5% in density in the range of interest.
- 3. Deviations and/or inconsistencies in the region of dissociation.

The last point proved to be especially troublesome. After equilibrium is established by dissociation, the properties presented should be those of normal hydrogen. The one variable which will reflect this change is

entropy; the amount of the change is 2 R ln 2. AiResearch or LASL tabulations do not reflect this change in entropy.

The next step will be to use the equation obtained for checking out Region 2 to improve the entries in the TAB code programs from 180° to 5000°R. This should eliminate all of the errors pointed out above.

W.J.H., R.D.M., H.M.R.

1.4 Equation of State for Parahydrogen

The equation of state last referred to in the 21st and 22nd Quarterly Reports has now been published in final form. The reference is "An Equation of State for Fluid Parahydrogen from the Triple-Point to 100°K at Pressures to 350 Atmospheres" by Robert D. Goodwin, J. Research NBS, 71A, 203-212 (May-June, 1967).

R.D.G

1.5 Absorption of Thermal Radiation in Liquid Hydrogen

The path length of the absorption cell has been increased to about 1". The pure rotational spectrum of liquid nitrogen has been obtained with this length. These observations, together with those for 1/2", enable a correction to be made for the reflection loss at the quartz-liquid interface and, hence, the true absorption in liquid nitrogen may be calculated. This calculation is now in progress.

The absorption spectrum of liquid methane has also been recorded in the wavelength range 40-500 microns. Absorption is stronger than for liquid nitrogen except at long wavelengths. The absorption spectrum of liquid normal hydrogen is to be recorded next.

M.C.J.

2. Cryogenic Properties of Solids

2.1 Thermal Conductivity of Solids

2. 1. 1 General Comments

The objective of this project is to determine the thermal conductivities of several aerospace alloys and standard reference materials from liquid helium temperatures to above 120°K.

Personnel contributing during the present reporting period are J. G. Hust and R. L. Powell.

2.1.2 Program Status

During the current reporting period several runs at liquid helium temperatures were accomplished. These were primarily for the purpose of "in place" thermocouple calibration, but also to further check out the operation of the apparatus. The Chromel vs. AuFe thermocouple attached to the same measuring station as the germanium resistance thermometer was calibrated at 109 points between 4 and 30°K. The resulting emf vs. temperature curve and the thermopower vs. temperature curve obtained from a smoothing of these data are illustrated in figures 2. 1. 1-2. Thermal and electrical conductivity measurements have also been conducted at liquid helium temperatures but due to the problems described in the last report these data are uncertain by several percent. These preliminary data are illustrated in fig. 2.1.3. The electrical resistivity of this sample in the liquid helium range is constant at 138.7 micro-ohm cm and the thermopower of the sample with respect to n-Ag varies from $0.82 \mu v/^{\circ} K$ at $13.7^{\circ} K$ to $1.65 \mu v/^{\circ} K$ at $24.5^{\circ} K$. The Lorenz ratio, $L = \rho \lambda / T$, is 13.9 x 10⁻⁸ watt ohm/° K² in this range. The new larger Ti alloy sample, redesigned thermocouple holders and sample heater have been fabricated and assembled. Measurements will proceed in the near future.

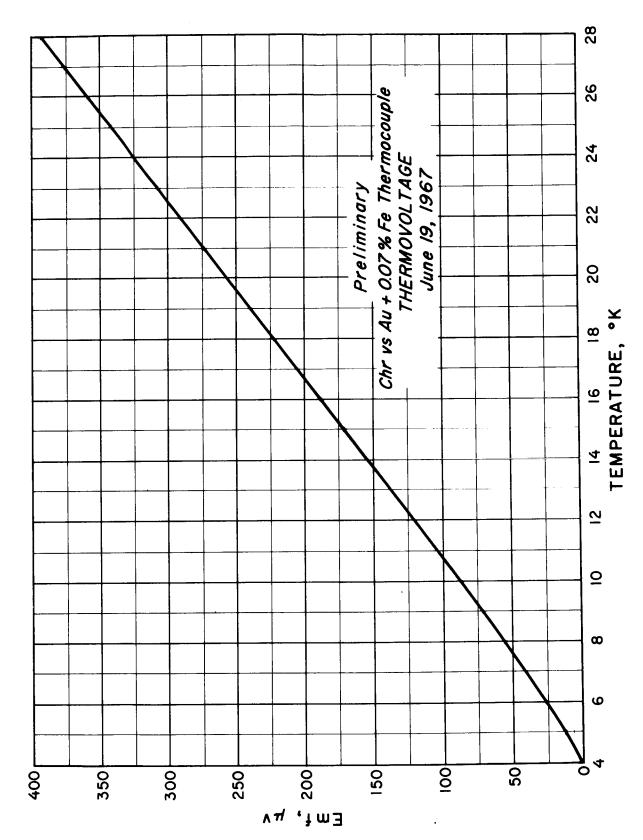
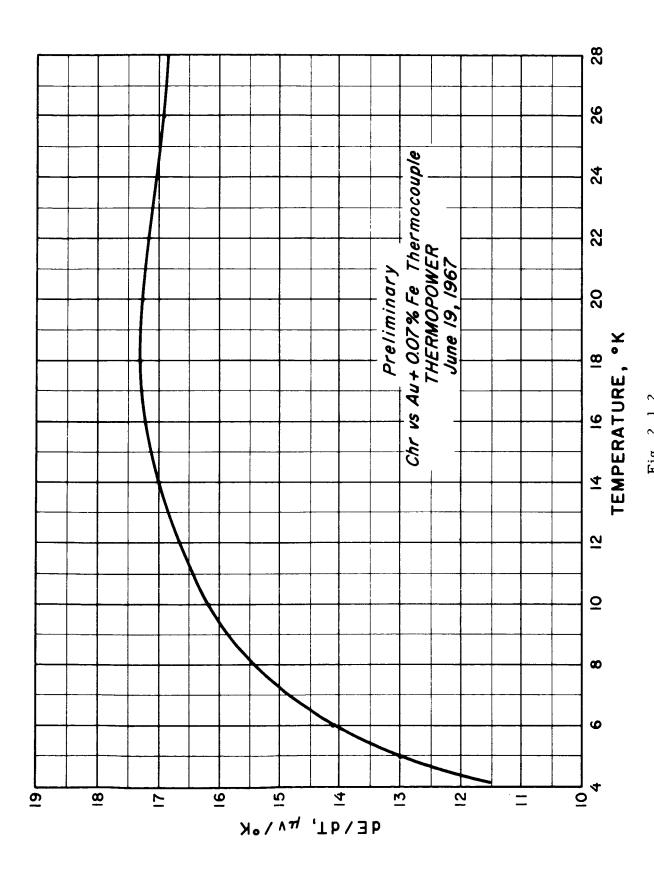
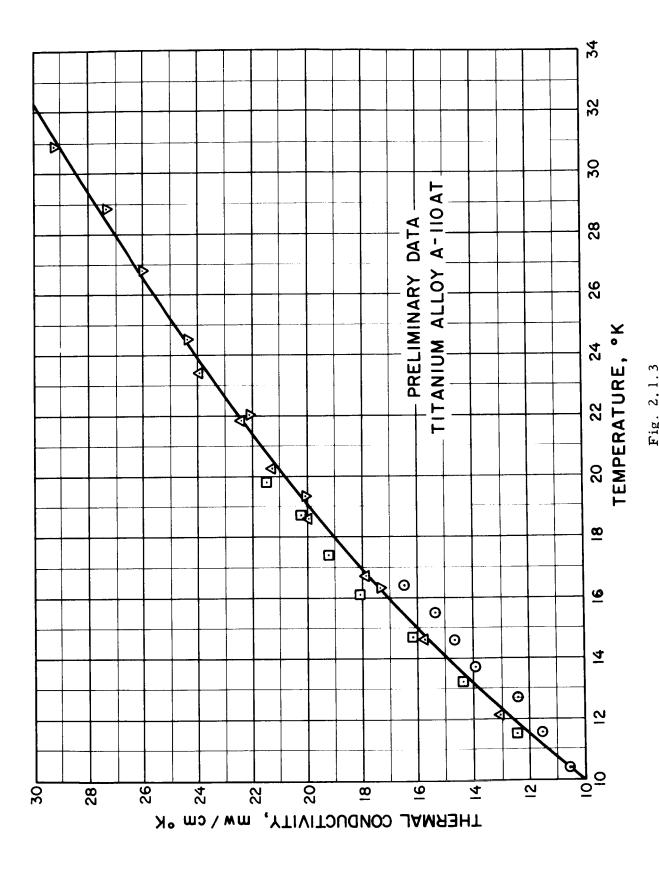


Fig. 2.1





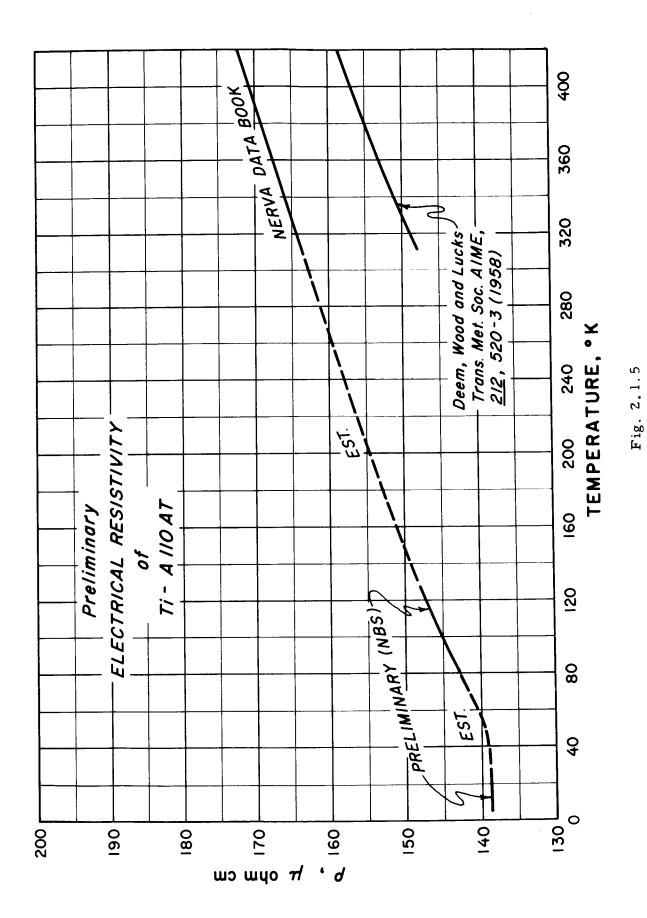
During the current reporting period approximately six weeks were lost on this program because J. G. Hust was required to complete the task of temperature scale comparisons according to a previous commitment to the Cryogenic Data Center. Also considerable consultation with the General Dynamics/Fort Worth thermal conductivity radiation effects program occurred during this period.

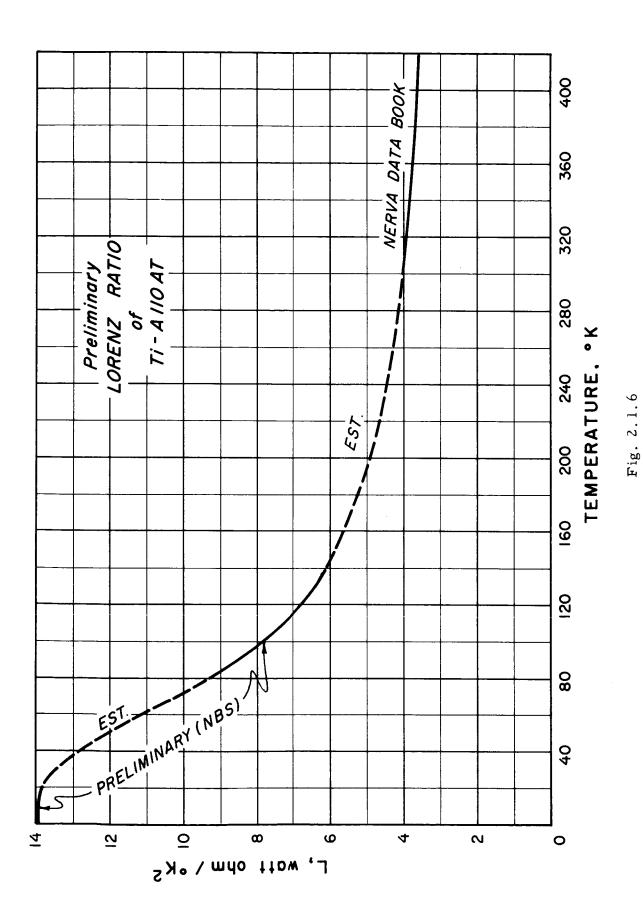
During the next reporting period, measurements on Ti-A 110 and Inconel 718 should be completed.

2.1.3 Materials Properties Data Book

The preliminary thermal conductivity, electrical resistivity, and Lorenz ratio of Ti-A-110AT alloy are presented in figures 2.1.4, 2.1.5 and 2.1.6. The uncertainties of the preliminary NBS measured values of thermal conductivity are relatively high because of the low conductance of the small sample used. Future measurements on this Ti alloy will be performed on a sample having ten times the cross-sectional area. The electrical resistivity shown in the NERVA Data Book represents a reasonable extension of the NBS measured values. The disagreement of these data from those of Deem, Woods and Lucks has not been satisfactorily explained at present.

Fig. 2.1.4





2.2 Thermocouple Thermometry

2.2.1 General Comments

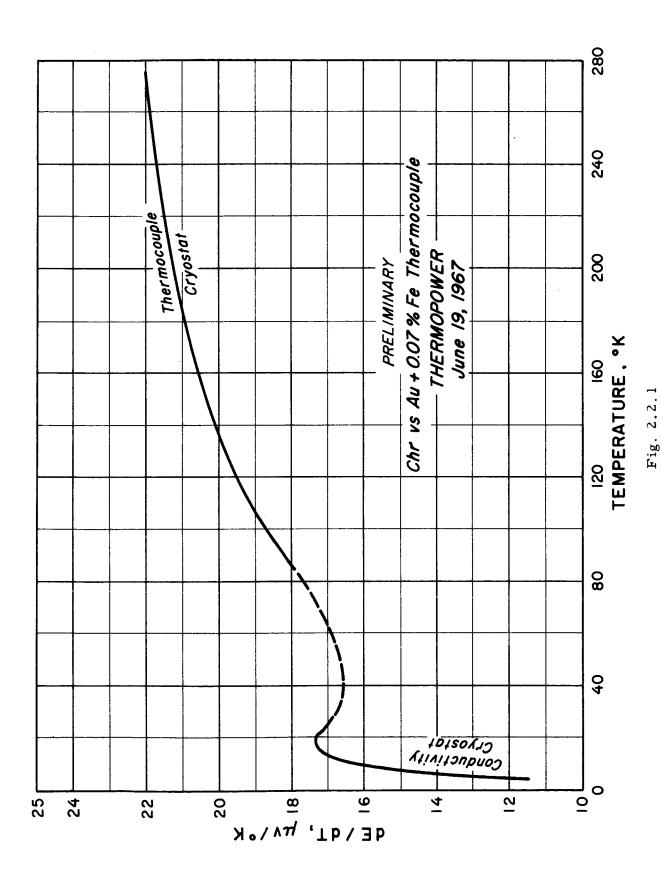
The objectives of the thermometry program are 1) to develop better thermocouple materials for the liquid hydrogen temperature range, and 2) to produce national standard thermocouple tables for materials used in the cryogenic-to-room temperature range. The present set of wires being calibrated include Chromel, Alumel, Constantan, copper, normal silver, and platinum. Materials in a development stage include silver-gold and three gold-iron alloys.

Personnel contributing during this reporting period were L. L. Sparks and R. L. Powell.

2.2.2 Program Status

Calibration has been completed in the liquid nitrogen temperature range (75°K to 290°K). "Hand" calculations, which include no corrections for the potentiometer, Mueller bridge, or temperature drift, indicate a measurement inaccuracy of from 0.03 to 0.07 µv in the thermocouple emf measurements. They also tentatively indicate the major error in the dependent variable (thermocouple emf) to be caused by the six dial potentiometer. The computer program which will ultimately be used to analyze all of the data will permit a very thorough analysis of error.

Fig.2.2.1 is a composite thermopower vs. temperature curve for Chromel vs. Au-0.07 atomic percent Fe. The portion of the curve from 4°K to 30°K was taken from the thermal conductivity experiment while the portion from 80°K to 280°K was accomplished in the thermocouple calibration apparatus. This portion of the curve was made up using nominal temperatures and with no corrections to the output voltages. It should, therefore, be considered preliminary. On the compacted scale used, however, it should be representative of the final form. The portion of the curve derived from the thermal conductivity apparatus will be



re-done in the more accurate thermocouple calibration apparatus; the portion shown by dashed lines will be included in the liquid hydrogen range thermocouple calibration.

The previously mentioned computer program is nearly completed in first draft form. When completed, the program will compare the thermoelectric output from each thermocouple wire to all similar wires, to each set of reference wires (platinum and normal silver), and to each of the dissimilar wires that it can be paired with. These intercomparisons will allow us to select equations which will minimize errors and give the best functional representations.

At the present time a vacuum leak occurs when the calibration cryostat is below the temperature of liquid nitrogen. The leak is almost certainly due to thermal contractions generated below 75°K — the system showed no leak at 75°K.

2.2.3 Summary

The liquid nitrogen range calibrations have been completed. During these runs the measurement system, control system, and vacuum system performed very well. As soon as the vacuum system repair is completed, the liquid helium range calibrations will commence. These runs are expected to take approximately one week. The liquid hydrogen range calibration will follow the helium tests and should take two weeks to complete. The computer analysis program is progressing well and should be completed by mid-July.

2.3 Thermal Expansion

2.3.1 General Comments

The objective of this project is to measure the thermal expansion coefficients of several aerospace alloys and standard reference materials from liquid hydrogen temperature, 20°K, to room temperature, 293°K.

The dilatometer apparatus is fully operational and calibrated with OFHC polycrystalline copper. The first sample, Hastelloy X, has been measured and is reported. Compilation and critical evaluation of data taken from the literature is in progress, with some initial results tabulated in this report.

Personnel contributing during this period were A. F. Clark and W. J. Hall.

2.3.2 Program Status

The apparatus was cleaned, repaired, and reassembled. Changes were incorporated in the cryogenic fluid venting system to comply with liquid hydrogen safety procedures. Also for safety reasons, glass components were replaced with stainless steel wherever possible. Chromel-Constantan thermocouples were placed on a sample and calibrated following NBS Report 8750[1]. A complete schematic of the dilatometer apparatus is shown in figure 2.3.1. The apparatus is now operational and the only further changes will be to automate the data acquisition. A linear differential transformer will replace the existing dial indicator, and its signal along with the signal from the thermocouple will be fed into a digital voltmeter and printed automatically.

Samples have been machined and ground to the dimensions 8.000" ± 0.002" long and approximately 1/4" square, with squareness to ± 0.002". Any samples which where bent or worked during machining were discarded in order to assure a "commercially available" condition. All samples will be hardness tested before and after measurements.

The apparatus was calibrated by measuring the thermal expansion of polycrystalline OFHC copper machined from 3/4-inch bar stock. Two copper samples were measured, one of them several times,

^[1] L. L. Sparks and R. L. Powell, NBS Report 8750, February 1965.

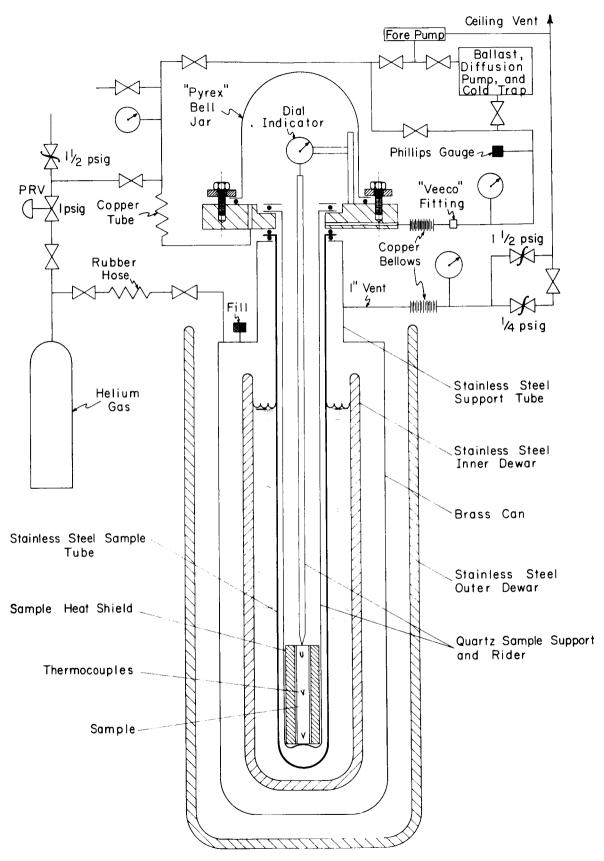


Fig. 2.3.1 Dilatometer Schematic

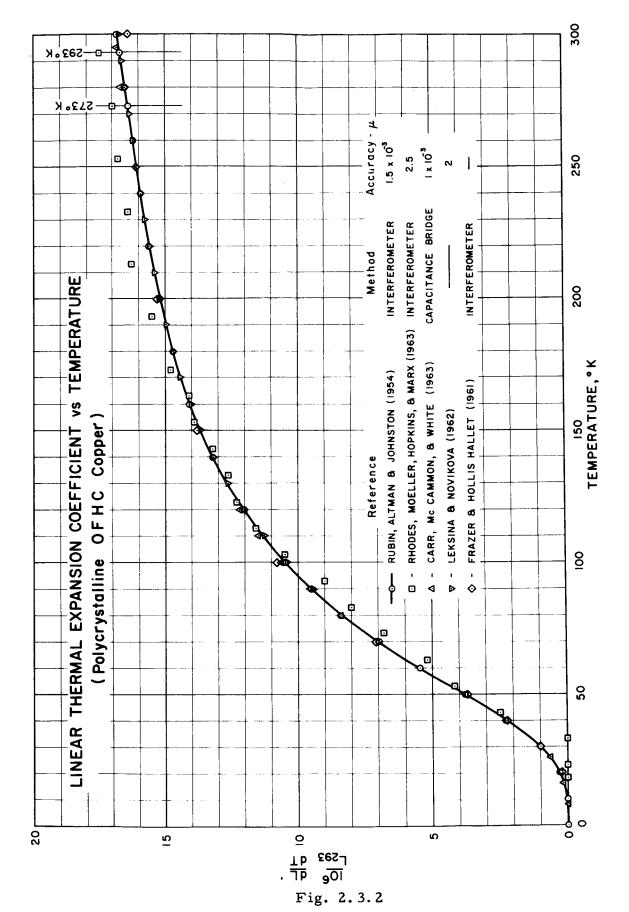
to determine reproducibility of the measurement on a given sample and between different samples. The largest variation of the total expansion between 293°K and 20°K was 1.2 percent and was usually better than 1 percent. The difference between an average of these data and the best literature data was then used as a correction to other sample measurements. The best literature data for copper were determined from figure 2.3.2 which shows the thermal expansion coefficient measurements of copper since the Corruccini and Gniewek[2] compilation of 1961. The Rubin, Altman, and Johnston[3] data were determined as "best" and used for this calibration.

Further measurements of copper were done to determine the smoothness of fit of data taken above and below liquid nitrogen temperature, and the effect of the cooling rate on temperature gradients in the sample. Figure 2.3.3 shows the smooth transition of data taken using liquid nitrogen as a coolant to those using liquid hydrogen as coolant. Proper placement of thermocouples will assure average temperature determination to better than 0.5°K for samples of low thermal diffusivity for the highest cooling rates.

Hastelloy X was measured and corrected as described and its total thermal expansion (referred to room temperature) and differential expansivity is shown in Table 2.3.1. The 90 percent confidence interval for the expansion values ($\Delta L/L$) tabulated is 2 x 10⁻⁵ or 2 units in the table. The differential expansivities are derived from the total expansion and may have a larger error. (The inaccuracies of the computer curve fitting show up as a decrease in the differential expansivity near room temperature, which is probably not real.) A more detailed discussion of errors will be given in the next report.

^[2] R. J. Corruccini and J. J. Gniewek, NBS Monograph 29, May 1961.

^[3] T. Rubin, H. W. Altman, and H. L. Johnston, J. Amer. Chem. Soc. 76, 5289 (1954).



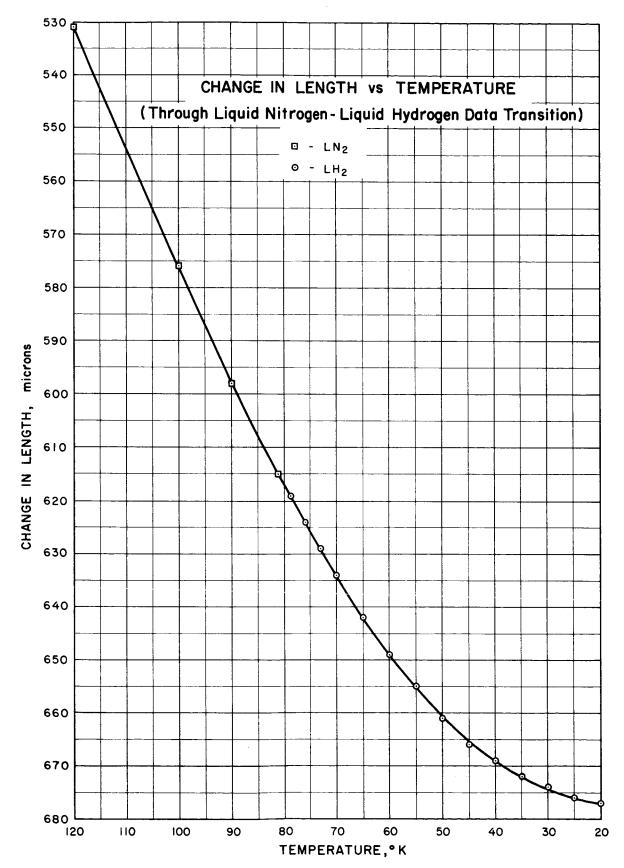


Fig. 2.3.3

A "Tymshare" computer program to convert existing literature data to a standard tabular form has been developed. From tabular values of thermal expansion referenced to any temperature and in any temperature scale, it 1) converts these to the Kelvin temperature scale referenced to room temperature, 2) fits the data with a five term polynomial in temperature, and 3) determines the expansion and expansivity at a standard set of temperatures. As shown in the Hastelloy X data, it can also be used to fit the experimental measurements. Literature data on the thermal expansion for some materials are tabulated in section 2.3.3 of the report.

The literature survey, done for the selection of test materials, has led to several conclusions:

- 1. A critical evaluation and compilation of the thermal expansion of solids at low temperatures should be done. The last such compilation[2] was primarily of the elements and does not include many alloys.
- 2. Thermal expansion measurements should be made of many of the newer nickel and magnesium alloys.
- 3. A series of related alloys should be measured to determine the dependence of thermal expansion on large compositional variations, e.g., iron-nickel from pure iron to pure nickel.
- 4. A typical alloy (or alloys) should be measured to determine the dependence of the thermal expansion on its heat treatment or condition. A good choice would be one of the more common high strength aluminum alloys.

Tests to cover these points are planned in this program.

Comparison of literature data for alloys of similar composition, such as the 5000 or 7000 series of aluminum alloys or the carbon steels, shows that for small variations (a few percent), little change can

be expected in the thermal expansion. For example, the work of Dorsey[4] shows that the difference in the average expansion coefficient from room temperature to 90°K between 1020 and 1025 carbon steel will be less than 0.5 percent. Also, the variation of the expansions from 293°K to 20°K for nearly all of the high strength aluminum alloys is only about 3 percent. These factors will be considered in the final selection of materials.

In the next reporting period, the remaining samples will be acquired and machined, measurements will be performed on many of them, an error analysis will be done, and the data acquisition should be automated. Also, the computer program for the data compilation will be converted to the laboratory's computer, and the existing literature data compilation completed.

^[4] H. G. Dorsey, Phys. Rev., 30, 271.

2.3.3 Materials Properties Data Book

The thermal expansion of Hastelloy X has been measured and is shown in table 2.3.1. The subsequent tables provide data missing from the Data Book. The appropriate references are given for each material.

Table 2, 3, 1 Linear Thermal Contraction and Coefficients of Linear Thermal Expansion

1	1	ı																									}
	n 7075	$\frac{10^6}{L_{293}} \frac{dL}{dT}$	deg 1 K	00.00	a0.25		1.96			6, 25	7.82	9.38	10.86	12, 23	14, 58	16.35	17.62	18.58	19, 45	20, 41	21.52	22, 58	22.99	23.01	22, 44	!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!	it al.
	Aluminum	10 ⁵ L293 - LT L293		a419	a419	418	417	414	410	405	398	389	379	368		310	276	239	201	162	120	92	46	30	0	1	Arp et al.
	ım 356	$\frac{10^6}{L_{293}}$ dL	deg ⁻¹ K	0.00	a0.23	0.85	1.79	2.97	4.32	5.77	7.27	8.75	10.19	11.53	13.87	15.66	16.92	17.77	18.39	18.95	19, 58	20.26	20.62	20.74	20.68	1 1 1	t al ^c
	Aluminum 356	10 ⁵ L293 - LT L293		a390	a390	390	388	386	382	377	371	363	353	342	317	287	255	220	184	146	108	89	41	2.7	0	1 1 1	Arp et
	oy X	$\frac{10^6}{L_{2.9.3}}$ $\frac{dL}{dT}$	deg ⁻¹ K	00.00	a0.14	0.51	1.07	1.76	2,56	3,41	4, 28	5, 14	5.97	6.75	8, 13	9,22	10.08	10.78	11, 41	12.02		12.83	12.64			i ! !	work ^b
	Hastelloy	10 ⁵ L293 - LT L293		a238	a238	238	237	235	233	230	226	222	216	210	195	177	158	137	115	92	29	42	25	16	0	1 1	This work ^b
		[-1	deg K	C	10	20	30	40	20	09	20	0 8	06	100	120	140	160	180	200	220	240	7 50	273	282	293	300	Ref.

Extrapolated

NBS Cryogenics Division, Data to be published. Hastelloy X condition; hot rolled, annealed, $R_{\tilde{c}} = 19$. V. Arp, J. H. Wilson, L. Winrich, and P. Sikora, Cryogenics 2, 1 (June 1962). င ဌာ

Table 2.3.1 (Continued)

	Steel, AISI	SI 1020	Steel, AISI	SI 4340	Steel, AI	AISI 303
[-	10 ⁵ L293 - LT L293	10 ⁶ dL Le ₉₃ dT	10 ⁵ L293 - LT L293	10 ⁶ dL L293 dT	10 ⁵ L293 - LT L293	$\frac{10^6}{L_{293}} \frac{dL}{dT}$
deg K		deg 1 K		deg 1 K		deg ⁻¹ K
0	a202	00.00	a198	00.00	a301	0.00
10	a202	1	a198	a0.09	a301	a0.17
20	202	a0.1	198	0.34	301	0.63
30	201	.3	197		300	1.33
40	201	8.	196	1.23	298	2.22
50	200	1.4	195	1.81	295	
09	198	2,3	192	2.44	262	
200	195		190	3.12	287	5, 52
- X	192	4.0	186	3.81	281	
06	187	4.8	182	4.50	273	
	182		177	5, 17	265	
130	120	ຸ່	166		245	
120			152		222	12.27
140	138		136		196	
180	120		118	9.12	169	13.85
	101	σ	66	9.68	141	14.14
330	101		62		113	14,35
340		10.1	59		84	14.68
07.6	27.0		37	10.96	54	15.37
007			23	11 20	33	16.12
273	22.9	11.4	67,	11.20	22	9
280	15.1	11,5	IS	•	11 (1)	;
293	0.0	11.7	0	11.50	0	68.71
300	-8.3	11.9	a -8	all.56	a-13	a18.64
))						
Ref.	Corruccini a	Corruccini and Gniewek	Arp et al	et al.	Arp et al	et al.

Extrapolated R. J. Corruccini and J. J. Gniewek, NBS Monograph 29, May 1961.

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Table 2.3.1 (Continued)

E	Steel, Al	AISI 316	Steel, AIS	AISI 630 ^e	Ti-5Al-2.	2.5Sn ^f
≒	10 ⁵ Lega - LT Lega	$\frac{10^6}{L_{2.9.3}}$ $\frac{\mathrm{dL}}{\mathrm{dT}}$	$10^5 \frac{\text{Lz93} - \text{L}}{\text{Lz93}}$	$\frac{10^6}{L_{293}}$ $\frac{\mathrm{dL}}{\mathrm{dT}}$	10 ⁵ L293 - LT	$\frac{10^6}{L_{293}}$ $\frac{dL}{dT}$
deg K		deg ⁻¹ K		deg ⁻¹ K		deg ⁻¹ K
0	a297	00.00	a197	00.00	a174	00.00
10	297	a0.04	a197	a0.12	a174	a0.10
20	297	a0.09	197	0.43	174	
30	297	0.5	196	0.91	173	0.79
40	762	1.4	195	1.49	172	1.30
50	294	2.7	193		171	1.89
09	290	4.3	190	2.85	169	2,51
20	285	6.5	187		166	3, 16
80	277	8.2	183		162	3, 79
06	569	9.4	179		158	4, 40
100	259	10.2	173		153	4.98
120	23.7	11.3	161	6.50	142	5.97
140	214	12.1	148		130	6.76
160	189	12.7	133	7.82	116	7.36
180	163	13.2	116		100	7.84
200	136	13.6	66	8.88	84	8.27
220	109	14.1	81	9.58	29	8.70
240	80.1	14.5	61	10.44	49	9, 14
790	9.09	15.0	39	11.34	31	9,46
273	30.9	15.3	24	11.77	18	9.48
280	20.2	15.4	16	11.89	12	
293	0.0	15.7	0	11.77	0	8.89
300	- 11.0	15.8	м Ф-	all.44	: !	!
Ref.	Corruccini and Gniewek	and Gniewek	Arp et al.	et al.	Arp et al.	t al.

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Extrapolated Stainless Steel 17-4PH Titanium A-110-AT

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Table 2.3.1 (Continued)

	Ti-6AL-4VB	t-4vg	Molybdenum	enum	Tantalum	lum
Ŧ	10 ⁵ Less - LT Less	10 ⁶ dL L ₂₉₃ dT	10 ⁵ 1293 - LT L293	10 ⁶ dL L293 dT	10 ⁵ L293-LT L293	10 ⁶ d1, L25 c dT
deg K		deg ⁻¹ K		deg ⁻¹ K		deg ⁻¹ K
0	a173	0.00	a95	00.00	a143	00.00
10	7	a0.10		a0.01	a143	a0.05
20	173	0.37	a94.8	a0.06	a143	
30	173	0.78	a94.7	a0.2	a142	
40	172	1.30	a94.4	a0.5	a141	a2.0
50	170	1.91	a93.8	a0.8	a138	a2.8
09	168	2, 56			a135	a3, 5
20	165	3, 25	a91.3		a131	a4.1
80	161	3,94	89.4	a2.1	a127	a4.5
06	157	4,62	87.1	2.48	122	a4.9
001	152	5.27	84. 4	2.80	117	5.2
22	140	6. 43	· ∞		106	
	127	7,33	71.1	3.77	95.1	5.8
160	111	7.96	63.2	4, 13		
180	56	8.32	54.6	4.42	71.5	0.9
000	28	8. 48	45, 6	4.63	59.3	6.1
220	61	8, 50	36.2	4.78	47.0	6.2
240	44	8.47	26.5	4.89	34.4	
260	27	4	16.6	4.98	21.6	6.5
273	٦,	4	10, 1	5.02	13.1	
000	2 -	4	9.9	5.04	8.5	
203	2	8.52	0.0	5.07	0.0	9.9
300	a -7	ici	-3.6	5.09	-4.6	9.9
))						
Ref.	Arp,	Arp, et al.	Corruccini	Corruccini and Gniewek	Corruccini	Corruccini and Gniewek
	ı					

Extrapolated Titanium C-120-AV e 90

Table 2.3.1 (Continued)

	Tungsten	sten	Copper	10		
Ŧ	10 ⁵ L293 - LT L293	10 ⁶ dL L293 dT	10 ⁵ L293 - LT L293	$\frac{10^6}{L_{293}} \frac{dL}{dT}$	10° L293- LT L293	$\frac{10^6}{L_{293}} \frac{dL}{dT}$
deg K		deg"1 K		dcg ⁻¹ K		deg ⁻¹ K
0	a85.8	00.00	a326	0.00		
10	a85.8	a0.007	a326	a0.04		
20	a85.8	a0.06	326	0.3		
30		a0.2	325	1.0		
40	_	a0.6	324	2.3		
50	a84.5	al.0	321	3.8		
09	8	al, 5	316	5.5		
70		al.8	310	7.0		
80	a79.7	a2.2	302	8.4		
06	a77.4	a2,4	293	9.5		
ć.	0 770	7 6	283	10 7.		
		- 6	0.76	0 0 0		
071	1.69	3,00	200	12.0		
140	62.7	3, 38	235	13.4		
160	55.6	3,66	807	_		
180	48.1	3,89	179	14.7		
200	40, 1	4.07	149	15.2		
220	31.9	4, 20	118	15.6		
240	23.4	4, 30	87	15.9		
260	14.7	4,39	55	16.2		
273	6.8	4,44	33	16.4		
280	5,8	4, 46	22	16.5		
293	0.0	4, 49	0	16.7		
300	-3.2	4.52	-11	16.8		
Ref.	Corruccini	Corruccini and Gniewek	Corruccini	Corruccini and Gniewek		
,						

3. Consultation and Advisory Services

3.0 General Comments

Consultation and advisory services in the general field of cryogenic engineering have continued in several NASA program areas:

Centaur (funded separately) and NERVA.

3.1 Centaur Program - Robert W. Arnett

Contact has been maintained with personnel at the Centaur Project Office, NASA-LeRC, through telephone conversations and mail communications.

3.1.1 Stratification and Pressurization

Minor alterations have been made in the computer program to ensure consistency with the developed equations. Negligible change in the computed values resulted from these alterations. A series of computations have been made with input parameters based upon reports of test program results from the literature. Three of the reports presented experimental data of Tatom and Hines and one presented Pt. Loma lockup tests conducted in October 1963 on a Centaur tank. These comparisons show a more gradual variation in temperature in the stratified region for the computer program than that obtained during the reported tests. Rather sharp breaks in temperature occur in the test results with either a poorly defined lower boundary of the stratified layer, or a much thinner stratified layer than predicted from the computer program. Work is continuing to determine the reason(s) for the observed differences.

3.1.2 Flight Data Analysis

Analysis of the AC-9 flight data was completed during the reporting period. The flight was very nominal in essentially all areas with no significant variations from evaluations previously made by NASA-LeRC. Since no particular differences were observed, an

informal letter report was supplied to the Centaur management together with the flight data curves prepared at this laboratory. This specific task is considered completed.

3.1.3 Helium Facility Study

Operation of the helium recovery system at Complex 36B, KSC, has been acceptable. Occasional shut downs due to a variety of causes are gradually being reduced as operating experience is gained. Since these are operational and maintenance problems at present, this task is considered to be completed.

3.2 NERVA Program - Alan F. Schmidt, Daniel H. Weitzel

Throughout the quarter, considerable exchange of information occurred by letter, telephone and meeting in regard to the thermal conductivity radiation effects program. On April 14, personnel from Aerojet-General Corporation met in Boulder for detailed discussions and presentations by NBS on thermal conductivity experimental error source and error analysis considerations, data acquisition and handling methods, and related items of interest. Subsequent to this meeting, an AGC (rough draft) experimental design plan was submitted to NBS for critical review. On May 18-19, the Third Radiation Effects Program Meeting was attended at General Dynamics/Fort Worth where current program problems were discussed, and an extremely tight program plan was developed to permit irradiation testing by mid-September. In order to receive benefit of NBS critical evaluation concerning the thermal conductivity experimental design plan (draft), the AGC project engineer met with NBS personnel in Boulder on June 1 to cover the business in detail. Several weeks after this meeting, the electrical resistivity experimental design plan was submitted to NBS for review and comments. On June 29-30, a meeting was held at GD/FW to discuss current status of the thermal conductivity and electrical resistivity

programs, to review non-irradiated experimental results achieved to date (with associated data handling techniques), and to resolve outstanding problems. A separate review was made of the para-to-ortho hydrogen conversion experiment at this time.

Also during the quarter, recommendations were given (at the request of SNPO-C) on (1) the suitability of several different types of cryogenic flowmeters at the NRDS Test Cell C, and (2) various questions concerning the applicability, use, and interpretation of results from instrumentation being employed on the Plum Brook Reactor Facility cryogenic helium flow loop.

3.3 Hydrogen Contamination - R. O. Voth, R. W. Arnett

During the current reporting period, contamination of liquid hydrogen with solid nitrogen was observed and photographed. Design of the test apparatus was completed and construction initiated.

Preliminary observations of solid nitrogen in liquid hydrogen were made using the slush hydrogen experimental apparatus. The settling characteristics of solid nitrogen were found to be dependent upon the method of contamination. Introduction of gaseous nitrogen to the hydrogen vapor (ullage) space, and subsequent condensation, caused the solid to settle rapidly (within approximately ten minutes). However, introduction of nitrogen below the surface of the liquid permitted the solid to remain in suspension for long periods of time (up to one hour). A definite reason for the difference in settling times is not known, but it is considered possible that it may be due to the presence of electrostatic charge on the particles in the latter case. When the nitrogen was introduced below the surface of the liquid and then stirred, settling occurred immediately. This event was recorded on 16 mm motion picture film.

The test apparatus for the forthcoming experimentation consists of a 300 $\,\mathrm{cm}^3$ expendable glass vessel suspended within a 1200

liter outer vessel. The 1200 liter shell is spherical in shape with 4 windows to observe the experiment; it will be evacuated before each test. The glass container and an associated liquid nitrogen shield are suspended from a top plate. The glass vessel contains an ignition source, a carbon resistor to measure liquid level, and a tube to introduce the contaminate.

A hot wire was chosen for the ignition source, and to be assured that sufficient energy is present to initiate a reaction, a spark gap of 3 mm has been established. Since the reported breakdown voltage for liquid hydrogen is on the order of 10 volts/cm, a 300,000 volt power supply is required. Later tests will determine if lower voltages can initiate a reaction.

Contamination of the liquid hydrogen will be accomplished by pressurizing an external vessel with the desired mixture of oxygen and nitrogen and allowing this mixture to flow into the liquid hydrogen glass test container. The liquid hydrogen will be used to condense the mixture, leaving only sufficient hydrogen to afford a short waiting period before the reaction is initiated. The apparatus is designed to allow the gas to be injected either above or below the surface of the liquid hydrogen. Tests will be conducted remotely with closed circuit television being used for observation.

During the next reporting period, construction of the apparatus will be completed and initial tests are planned.

4. Cryogenic Flow Processes

4.0 General Comments

Personnel contributing to the project during the present reporting period were J. A. Brennan and W. G. Steward.

4.1 Experimental and Analytical Transfer Line Cooldown

A series of tests was completed with the 1/4-inch O.D. transfer line including four driving pressures for subcooled and saturated liquid nitrogen and subcooled liquid hydrogen. The 1/4-inch line produced small surges in subcooled liquid nitrogen and practically no surging in hydrogen. Complete data for these runs will be shown in the final report now in progress.

The computation time has improved to the extent that a complete cooldown calculation for hydrogen now requires less than 45 minutes. A means of lumping segments containing pure liquid in a cold region of pipe permits smaller division of the fluid in the region of rapid change. Thus, better accuracy is possible with less computation.

A final report on transfer line cooldown is expected to be in draft form by the end of the next reporting period.

- 5. Cryogenic Propellant Venting Under Low Pressure Conditions
- 5.0 General Comments

Personnel contributing during this period were M. C. Jones and P. J. Giarratano.

5.1 Accomplishments during Current Reporting Period and Status of Project

Experimental heat transfer measurements for hydrogen as the test fluid have been completed and data reduction for both hydrogen and nitrogen is presently being accomplished using a CDC 3600 digital computer.

During the next reporting period data reduction will be completed and work will begin on presentation of results in a final report.

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